TRANSIENT LIQUID PHASE BONDING PROCESSES

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Abstract

Transient liquid phase (TLP) bonding is an ancient process that has received increased attention in recent years. It is capable of producing nearly invisible joints that have strengths and other properties similar to the base metal. In application, a thin interlayer material, such as a braze foil, is clamped at the faying surface and the entire assembly is brought to an elevated temperature. Bonding results as melting point depressants in the liquid interlayer diffuse into the metal which results in isothermal solidification. The concept dates at least as far back as the Etruscans who used a copper interlayer to join gold objects. More recently, TLP has been applied to joining nickel base superalloys, titanium, boron-aluminum composites and semi-conductor materials. The interlayer composition is selected to minimize bonding time, provide adequate wetting and avoid the formation of intermetallics. In this paper, a review of TLP is presented that emphasizes the practical aspects that are considered when designing the process for a given system. As well, the present theoretical understanding is discussed with a description of future research goals.

Introduction

Transient Liquid Phase (TLP) joints are formed when a melting point depressant (MPD) in the interlayer diffuses into the surrounding bulk and isothermal solidification results. The kinetics of the process are controlled by solid state diffusion into the material to be joined. The process has been applied to several metallic systems but the concept is not limited to any particular class of materials, but rather to systems whereby a chemical or other driving force inherently leads to solid state equilibrium. In this article, the term TLP will apply to those bonding processes which rely on solid state diffusion to drive the interlayer to solidification.

The TLP process has been divided into four stages by Tuah-Poku et al [1], dissolution, widening, isothermal solidification and homogenization as described in Fig. 1 A fifth stage termed stage 0 has been included here to account for the effects of a less than instantaneous heating rate, as discussed by Niemann and Garrett [2] and Nakagawa et al [3].

The rationale for this description follows from consideration of a binary eutectic diagram, as shown in Fig. 2. A layer of pure metal B is sandwiched between a structure of metal A. Upon heating to the bonding temperature the interlayer and parent

metal undergo interdiffusion to form a liquid phase. As the dissolution progresses, the liquid composition moves from C_{BL} to C_{LB}. The time required for this step has been estimated to be on the order of seconds [1]. This suggests that even if there had been prior interdiffusion, this step would dissolve the surrounding material remelting the MPD that had previously diffused away. As discussed later, this may not be trivial when one is designing interlayers close to the liquid composition of $C_{L\alpha}$. Upon the completion of the dissolution stage, the widening of the interlayer drives the composition to the alpha rich
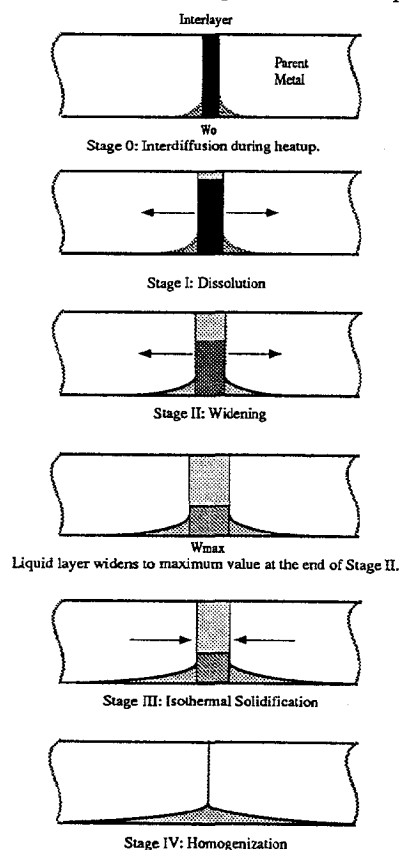

Figure 1: Four stages of TLP Bonding as defined by Tuah-Poku et al [1]. Stage 0 has been included to account for interdiffusion occuring during heatup. The grey shading indicates the concentration of the melting point depressant (MPD). The arrows indicate the direction of movement of the solid-liquid interface.
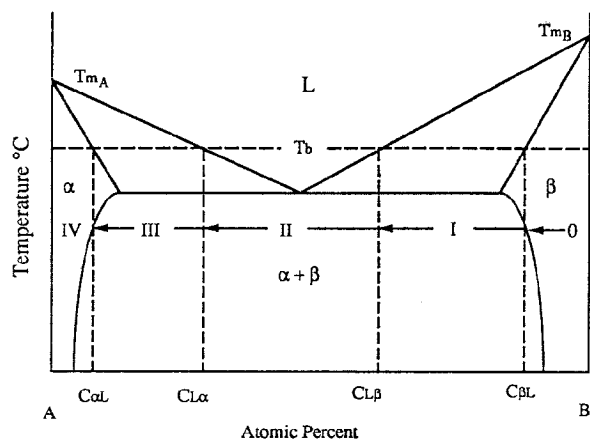
Figure 2: Schematic of a binary eutectic phase diagram showing the stages in transient liquid phase bonding. An interlayer of B rapidly dissolves the parent metal A until the composition at $C_{L\beta}$ is reached. Widening next occurs lowering the concentration of B in the liquid to $C_{L\alpha}$. Isothermal solidification then reduces the amount of liquid although the composition remains constant at $C_{L\alpha}$. Once solidification is complete, the maximum concentration in the joint can be reduced from $C_{\alpha L}$ by homogenization.

liquidus at $C_{L\alpha}$. The widening process requires times on the order of minutes as reported by Nakao et al [4] and Tuah-Poku [1].

Stage three or the isothermal solidification step is by far the most important step as it requires the greatest amount of time and is dependant on the width of the liquid interlayer formed and the rate of diffusion into the bulk. During this stage the diffusion of the MPD into the parent metal occurs at a rate dependant on the diffusion constant in the bulk, provided there are no kinetic restrictions at the interface. An interesting characteristic of this stage is that there exists a temperature at which a minimum in the solidification time occurs [1].

Homogenization in stage four completes the process and is dependant on the time at temperature. It is controlled by a solid state diffusion rate similar to other homogenization processes. The homogenization time is a function of the required maximum tolerable MPD concentration.

One criterion for the selection of TLP is whether it will work within a reasonable time on a particular system. Because it is an in situ melting process, large and complex shapes can be assembled and bonded in one operation rather than having to make several smaller joints. The advantages and disadvantages of the TLP process are shown in Table I.

Historical Development

In the following a brief description of TLP-type processes is presented. A more complete version including a discussion of the theoretical models will be published in the Annual Review of Material Science [5].

Table I   Characteristics of the TLP bonding process.

Advantages	Disadvantages
• base metal properties at joint • no interface remains after bonding • self homogenizing • low joint pressure required • intermetallic formation can be avoided • minimum surface preparation • large and complex shapes bonded simultaneously	• long bonding times (hours) • restricted to high temperatures, $T/T_m=0.6$ • fast diffusers required, preferably interstitial elements • rapid heatup required • close fit-up required • post bonding heat treatment for age hardening alloys is required

The joining of metals with a transient liquid interlayer dates back to ancient times. A method for attaching decorative gold beads to ceremonial articles, known a granulation, was described by Cellini [6] in the sixteenth century. In this process, copper oxide is painted on the article and then gold balls are attached. The article is then placed in a reducing flame which causes the oxide to reduce to copper and then react and form a eutectic with the gold which disappears by diffusion. Several objects that are thought to have been bonded using this process are shown in C.S. Smith's book on archeometallurgy [7].

More recently, the emphasis has been on bonding high temperature materials such as titanium and nickel base superalloys. The process is easily applicable to these and other high temperature materials because they are thermodynamically stable at the high temperatures required for fast diffusion rates. It should be noted that over the past thirty years there have been several variants of the process developed which are listed in Table 2. It is interesting to note that a new name is often created when similar principles are applied to a new material system.

The first report of a TLP joint in modern times is due to Lynch et al [8] who prepared interface free TLP joints in titanium using a nickel-copper interlayer. A series of micrographs in their paper showed the progressive dissolution of the interlayer and the eventual formation of a joint that was "effectively just a grain boundary". A similar concept was developed by Owczarski [9] for joining dissimilar metals. Here only stage I and II are involved since the process is not allowed to go to completion. By adjoining Zircaloy 2 to 304 stainless steel above $980^{\circ}C$, a eutectic is formed that progressively dissolves each metal. Low ductility is a result of intermetallic compounds formed at the interface. Bernstein [10] and Bernstein and Bartholomew [11] developed the Solid-Liquid Diffusion (SLID) process for joining semi-conductor components. The joints were not taken to complete isothermal solidification, as this was not the objective. Rather, each joint in a sequence of joining processes had to be made at a lower temperature than the previous, hence a partially completed TLP joint would benefit from any high temperature excursion as long as it was not above the melting point of the interlayer.

Table II TLP-Type Processes listed in chronological order.

Process Name	Author	Type	Alloy System	Insert	Bonding Temp.°C
Granulation	Cellini [6,7] 1568	I	Au	CuO	800
Eutectic Brazing	Lynch et al [8] 1959	I	Ti	Ni	>955
Eutectic Bonding	Owczarski et al [9] 1962	III	Zircaloy 2 to 304 Stainless	None	980
Solid-Liquid Interface Diffusion (SLID)	Bernstein [10,11] 1966	I	Semi-conductors	In, Au	<500
Activated Diffusion Bonding (Diffusion Welding)	Hoppin and Berry [12] 1970	II	Superalloy René 80	Ni + MPD	?
NOR-TI-BOND	Wu [13], Wells and Mikus [14] 1971	I	Titanium structural shapes	Cu	920-955
Diffusion Welding	Duvall et al [15] 1972	I	Udimet 700 superalloy	Ni-Co	1093-1171
Eutectic Bonding	Niemann and Garrett [18] 1974	I	Boron-Al composites and Ti to Al	Cu	537
Transient Liquid Phase (TLP)	Duvall et al [17] 1974 and Ikawa, Nakao and Tsai [23]	II	Nickel Superalloy	Ni-P,B	1100-1200
Liquid Interface Diffusion(LID)	Norris [19] 1986	II	Titanium alloys	Cu-Ni	900-982
Transient Insert Liquid Metal (TLIM) Diffusion Bonding	Nakao et al [4]	II	Nickel Superalloy	Ni-P,B	1100-1280
TLIM + Powder	Nakao et al [26]	II	Nickel Superalloy	Ni-P	1150

The TLP process was applied to aerospace materials by workers at aircraft engine manufacturers. Hoppin and Berry [12] working at the Aircraft Engine Group at General Electric developed Activated Diffusion Bonding for joining superalloys such as René 80. The Nor-Ti-Bond process, described by Wu [13], was developed at Northrop by Wells and Mikus [14] to join titanium structural shapes. The two step process used locally applied resistance heating to melt electrolytically deposited copper at the interface followed by a subsequent heat treatment. At Pratt and Whitney, Duvall et al [15] joined the superalloy Udimet 700 using a Ni-Co interlayer using a process patented by Owczarski et al [16]. In the following year the same group coined the term TLP. Duvall et al [17], used boron in a nickel foil as the MPD for joining a similar alloy. Niemann and Garrett [2,18] of McDonnell Douglas, developed Eutectic Bonding for joining boron-aluminum composites with a copper interlayer. They were also able to make titanium-aluminum joints. Liquid Interface Diffusion bonding was developed at Rohr Industries to bond honeycomb sandwich structures using copper-nickel interlayers [19].

Most of these processes can be separated into two distinct variants depending on the initial interlayer composition. In type I processes the interlayer is a pure metal and hence is the MPD. This type of interlayer requires all four stages including interdiffusion with the parent metal before liquefying. The extent of widening is maximized as the concentration of the MPD must be reduced from unity to the liquidus composition at $C_{l\alpha}$. In type II processes, an interlayer at or close to the liquidus composition is used. In this case, only stage III and IV occur and the amount of MPD for a given initial thickness is reduced. This approach, used in superalloy TLP bonding, requires bonding times on the order of minutes versus hours in the type I approach. Eutectic brazing may be considered a third variant, in that stages I and II occur, but the process is not taken to completion. If it were, complete dissolution of at least one side of the joint would result.

The basis of the TLP process can also be found in the work on Diffusion Solidification [20]. In this process, molten high carbon iron is poured into a crucible filled with low carbon steel shot and solidification proceeds as the carbon diffuses into the shot. TLP Sintering of ceramics and refractory metals [21,22] requires an MPD to diffuse into the surrounding compacted powder. Dental amalgams solidify isothermally in a similar manner.

TLP Joint Design

Base metal

In principle, most metals could be TLP bonded by a suitable interlayer. In order to make a 100% efficient joint, ie. one that possesses parent metal properties, considerable care has to be taken. The physical properties of the base metal that must be considered include the melting temperature, strength at temperature and the ability to diffuse away bonding elements. This latter requirement is mainly a function of temperature and the nature of the melting point depressant. These considerations obviously suggest that superalloys would be ideal candidates for TLP bonding. The intrinsic stability of the superalloy close to its melting temperature allows for rapid diffusion and hence quick bonding times. The process is further enhanced by the use of boron or phosphorus as the MPD.

Because of the high temperatures involved, oxidation and pickup of other elements is a factor, hence atmosphere control or a vacuum system is required. If the MPD volatilizes at the bonding temperature, a reduction in the time required for solidification may result. Contamination, especially in titanium systems, can also be an issue.

Faying Surface Preparation

The interface must be clean and free of heavy oxides to maintain mechanical properties. The behavior of the liquid metal may be enhanced by having a rough interface as it initially provides greater surface contact area for the dissolution process. Typical preparation would include a polishing step followed by degreasing. The interfaces have to conform to one another. As the typical interlayer width is on the order of a few thousandths of an inch, joint fit-up tolerances must be of similar dimensions.

Flux is not required on the faying surfaces when TLP bonding nickel and titanium. Copper TLP bonds using a silver interlayer develop porosity which can be eliminated with flux. In general, flux should be avoided as it can leave undesirable compounds at the interface.

Interlayer

The interlayer composition must meet the following criteria. At least one element, the melting point depressant (MPD), must have solubility in the base metal. The MPD must have a significant diffusivity at the bonding temperature to ensure reasonable bonding times. Finally, the elements in the interlayer must not be detrimental to the physical and mechanical properties of the base metal.

In practical applications it is desirable to have the interlayer composition as close to the liquidus at the bonding temperature as possible. Consider two foil interlayers, one pure metal B and the other an A-B alloy close to the liquidus composition. The amount of MPD that must diffuse in the pure metal case is

$$W_o(C_B - C_{L\alpha}) \; grams/cm^2 \qquad (1)$$

where C_B, the initial concentration of the MPD, is unity for a pure metal interlayer and W_o is the initial foil thickness. The solidification time, as derived by Tuah-Poku et al [1], is

$$t_3 = \frac{\pi W_o^2}{16 D_\alpha} \left(\frac{C_B}{C_{\alpha L}} \right)^2 \qquad (2)$$

which indicates that the bonding time varies as the square of the initial MPD concentration and foil width. As C_B is reduced to $C_{L\alpha}$, the amount of MPD approaches the minimum required to form a liquid phase. This equation illustrates the advantage of using electroplated alloy interlayers which minimize both thickness and MPD concentration.

The critical step in the formation of the liquid zone is the dissolution of the parent metal. Intermetallics which form may hinder the dissolution process. Where there is a high melting temperature intermetallic compound surrounded by two eutectics as in Fig. 3a, the intermetallic will form at bonding temperatures below T_I but above each eutectic temperature. The time required to reach the α-rich liquidus will be increased because of the time required to redissolve the intermetallic. It is not expected that the intermetallic will form a continuous sheet between the two liquid compositions. The formation of an intermetallic via a peritectic reaction as in Fig. 3b, will have no effect on the solidification time in a binary system. The MPD will diffuse away governed by the rate at which it diffuses in the α phase and not in the intermetallic. Consider the silver-titanium phase diagram in Fig. 4. If one were to use a silver foil to TLP bond titanium, any bonding temperature above 961°C would in principle behave as a TLP process. However, if intermetallics are to be avoided in the final joint the maximum concentration of silver has to be less than about 12%. This is true for binary systems only. In ternary or higher order systems the activity of the MPD must be less than the activity of the MPD in the intermetallic. Bonding temperatures therefore must be over 1031°C to avoid beta and gamma phases in the Ti-Ag system. At lower temperatures, extended homogenization times would be required to eliminate the intermetallic phase.
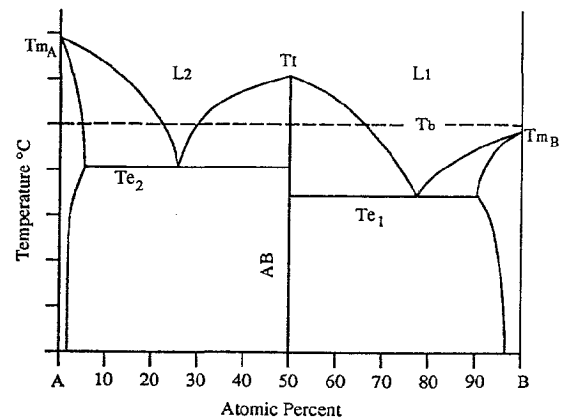

Figure 3a: Schematic phase diagram indicating the presence of an intermetallic phase which will hinder the kinetics of TLP bonding. At the bonding temperature, Tb, B liquifies, dissolves A and forms the intermetallic compound AB. To avoid this, the bonding temperature has to be raised above T_I.
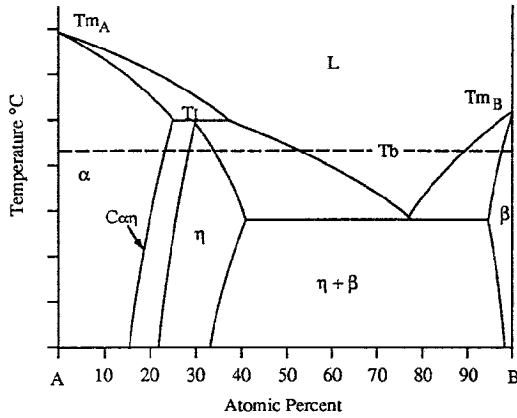
Figure 3b: Schematic of a binary phase diagram with a peritectic reaction. Solidification below Ti will not be constrained by the presence of the intermetallic η, but rather will depend on Cαη and the diffusivity of B in α.

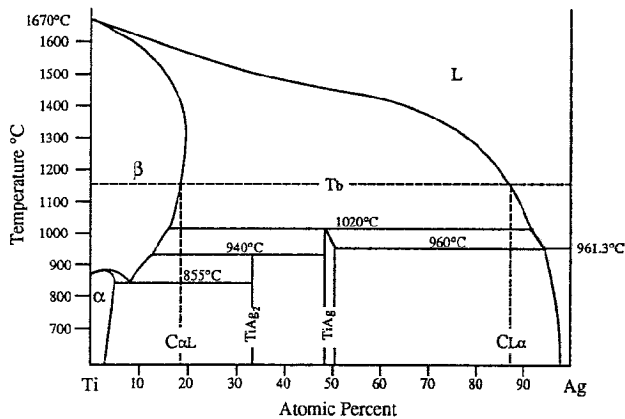Figure 4: Silver-titanium phase diagram. TLP bonding will occur above 961°C but the kinetics will be retarded by the formation of the intermetallicTiAg. At temperatures above 1031°C, the intermetallic will not form and the liquid composition $C_{L\alpha}$ will be in equilibrium with the solid solution phase at $C_{\alpha L}$.

If the MPD is a minor constituent of the interlayer and other constituents form an intermetallic with the parent metal, an increase in the bonding time will result. In this situation, the MPD has to diffuse through the intermetallic. Since diffusion in intermetallics is slow, the temperature must be raised above the melting point of the highest melting intermetallic in order to achieve rapid bonding times. The concentration path that the interlayer takes when solidifying is far more complex as it is dependant on the relative diffusivities and the phase relation between the elements. Consider TLP bonding of Cu with a Ag-10Au interlayer. On the ternary phase diagram sketched in Fig 5, the diffusion path is a straight line from the interlayer composition to the copper rich corner if the diffusivities of gold and copper are equal. However, the diffusivity of silver can be three times that of gold in copper depending on the bonding temperature. Thus once the copper rich liquidus is reached, the interlayer will enrich in gold until the corresponding increase in gold flux equals the silver flux. Since the temperature remains constant, the maximum possible gold concentration would

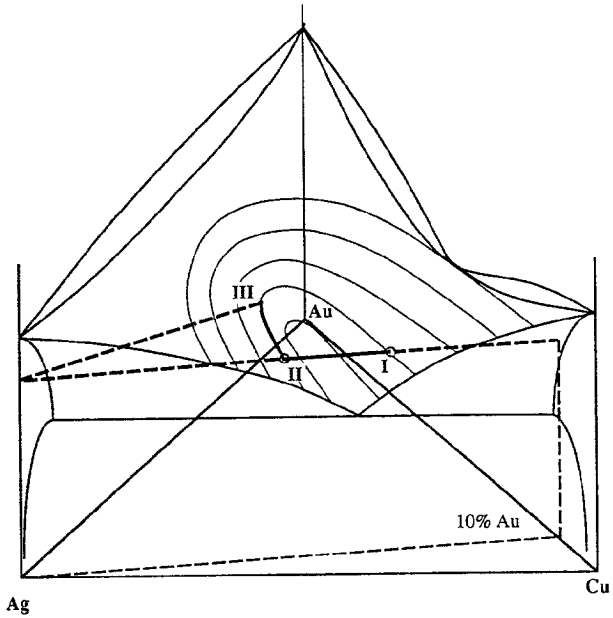Ag   Cu

Figure 5: Schematic ternary phase diagram for the silver-copper-gold system. Bonding with a Cu-10Au interlayer will form a liquid at I, which will quickly widen to II. Because of the difference in diffusivity, the interlayer will enrich in gold until the new concentration at III equilibrates the flux.

approach 50% as indicated on the phase diagram. By analogy, one would expect that in complex alloys with limited solubility of alloying elements, a complex solidification path would lead to intermetallic formation. Ikawa et al [23] showed that aluminum and chromium did not follow the calculated paths at early times when TLP bonding a nickel-base superalloy indicative of this phenomenon.

The interlayer can be provided in a variety of forms including foil, electroplate, sputter coat, powder, rod, paste, slurry and any other method that produces a thin film on the faying surfaces. The minimum thickness is determined from consideration of the heatup time and the amount of liquid needed to fully wet the interface. The amount of diffusion during heatup can be roughly estimated from the equivalent diffusion constant at temperature as given by equation (3) from Shewmon [24]

$$\bar{D} = \frac{\int_0^t D(t)\, dt}{t} \qquad (3)$$

The lower limit on the heating integral is taken as the time when the process temperature is 80% of the final bonding temperature, because at lower temperatures the contribution to the diffusive flux is negligible. Following Darken and Gurry's analysis [25], the average diffusion constant is then inserted into equation (4) which gives the coating loss as a function of heatup time,

$$X = KC_{\alpha L}(Dt)^2 \qquad (4)$$

97

where K is a constant dependent on the material system and $C_{L\alpha}$ is the solubility limit of the MPD in the parent metal. It is interesting to note that for a constant heating rate the average diffusion constant is independent of the rate. This can be shown by considering a relation for temperature as a function of time given by

$$T = \dot{T}t + 0.8T'$$ (5)

where T' is the bonding temperature and $\dot{T}$ is the heatup rate. The last term on the right restates that at $t=0$ the process temperature is 80% of the bonding temperature. This equation, along with the standard Arrhenius form of the diffusion constant, given by

$$D = D_o \exp(-Q/RT)$$ (6)

substituted into equation (3) gives

$$\frac{\overline{D}T'}{5\dot{T}} = \int_{.8T'}^{T'} D_o \exp(-Q/RT) \frac{dT}{\dot{T}}$$ (7)

From equation (7) it is apparent that the heatup rate cancels. The analytical solution of this integral unfortunately involves a slowly converging series function.

The powder method termed TLIM bonding is interesting because it not only provides the MPD but also a matrix into which to diffuse. In this method, the effective surface area available for diffusion is greatly increased by using a powder of roughly the same composition as the base metal. In this way, solidification times are reduced by over two orders of magnitude according to Nakao et al [26], hence bonding at lower temperatures is feasible.

Process Conditions

Stage III is the rate controlling step for the TLP process. Dissolution and widening occur on the order of seconds and minutes whereas isothermal solidification may require hours or days. As mentioned earlier, the kinetics of stage III are controlled by solid state diffusion into the parent metal.

The nomograph in Fig. 6 presents a method of determining the bonding time for a given diffusivity and liquidus composition. For a reasonable bonding time of 8 hours it is apparent that a diffusivity of 10^{-8} cm^2/sec with an initial foil thickness of 25 microns is required. The strong influence of foil thickness is revealed in Fig. 7 which is a plot of solidification time as a function of thickness and temperature for the copper-silver system. The minimum in the bonding time arises due to the trade off between the increase in the diffusivity and the amount of dissolution, which also increases with temperature [1].
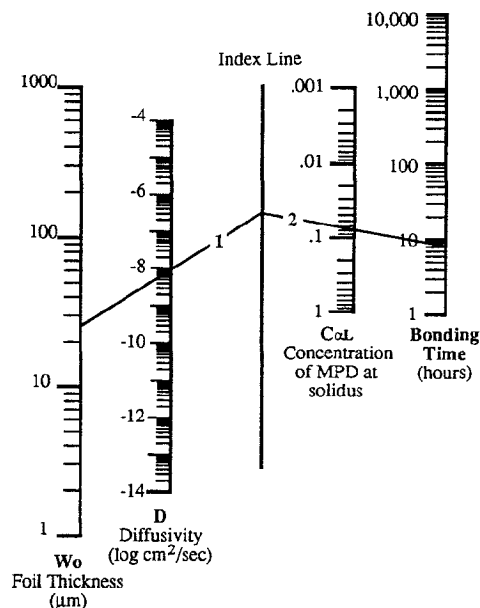

Figure 6: Nomograph based on equation (2) in text. To use, draw line 1 from the foil thickness through the diffusivity to a point on the index line. Then draw a line from that point through the concentration to obtain the solidification time. As an example, the result for a 25 μm (.001") foil with a diffusivity of 10^{-8} cm^2/sec and a concentration of .08 is shown. The resulting time is about 8 hrs.
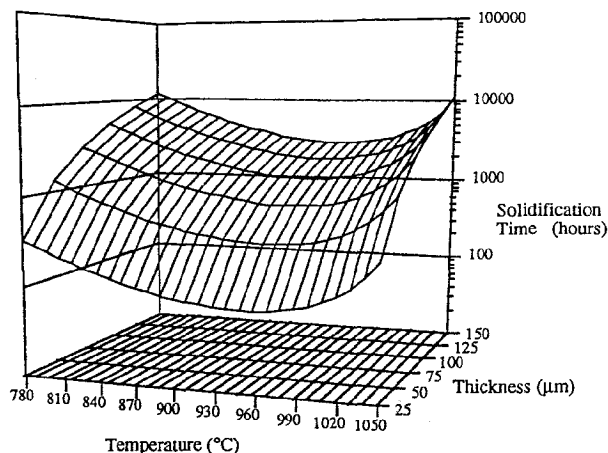

Figure 7: Isothermal solidification time for TLP bonding of copper using a silver foil. The minimum in the bonding time is at 955°C and is independant of foil width. Over the range of permissable bonding temperatures, the bonding time varies by less than an order of magnitude. Hence, a significant reduction in bonding time can only be achieved by reducing the foil thickness.

98

Joint requirements

One of the strongest claims for TLP is that it generates near parent metal strength and ductility. Ideally, the process would have no effect on the corrosion behavior, fatigue life, fracture toughness, thermal stability and other properties. However, there is much less published on these aspects of the process. Joint strength is often reported as a joint efficiency which is defined either as the percentage of the joint strength relative to the parent metal or the decrease in load required to achieve the parent metal stress rupture life.

Bernstein [10,11] developed an unbonding test for determining the extent of bonding. Samples were placed under shear and then heated until the joint failed. Two distinct ranges of unbonding temperatures were found using silver, gold or copper with indium interlayers. Most joints failed in the upper range of 500-800°C corresponding to melting temperatures of intermetallics in these systems. The lower range was below the bonding temperature of 300°C and occurred 10-15% of the time. These low unbonding temperatures were said to have been due to poor wetting, unsatisfactory mating of parts or some form of kinetic restriction, however, these results could be explained in the following way.

Consider SLID bonding using the silver-indium system shown in Fig. 8. During bonding at 300°C, dissolution of silver into indium continues until the liquidus composition is reached and intermetallics nucleate at the solid-liquid interface. If the growth is non-planar, dissolution of copper continues at a rate that replenishes the copper lost to the intermetallic. In this way, the intermetallic can bridge the interlayer and grow while dissolution continues. Thus, the unbonding temperature will depend strongly on whether the intermetallics have had sufficient time to bridge the gap. The variation in unbonding temperatures can be explained by the variation in the extent of bridging and volume of intermetallic. During the heatup, dissolution of the intermetallic by the remaining indium rich liquid will then accelerate the unbonding process. Evidence for the non-planar growth of the intermetallic is shown in Bernstein [12] and more recently by Roman [27].

TLP bonded nickel base superalloys have joint efficiencies of close to 100%. Hoppin and Berry [14] reported high temperature tensile, stress rupture and fatigue strengths on René 80 that were 70-90% efficient. Room temperature tensile tests were said to be misleading, as failure always occurred at the joint with a corresponding low ductility. Duvall et al [16,17] were able to join Udimet 700 and Hastelloy X without forming a brittle precipitate at the interface using a nickel-cobalt interlayer. Parent metal tensile and stress rupture strength requirements were met. Similar tests on IN-100 resulted in lower stress rupture properties and failure at the joint. This small set of data indicates that parent metal strengths can be achieved in nickel base superalloys, although the joint region is susceptible to embrittlement.

Mechanical properties of aluminum-boron composites using copper interlayers were discussed by Niemann and Garrett [18]. The design allowable strength, modulus and density were achieved. Since the material consisted of individual plies, a comparison to the base material strength was not feasible. The addition of one titanium ply for every four boron-aluminum plies increased the strength and the strain to failure by a factor
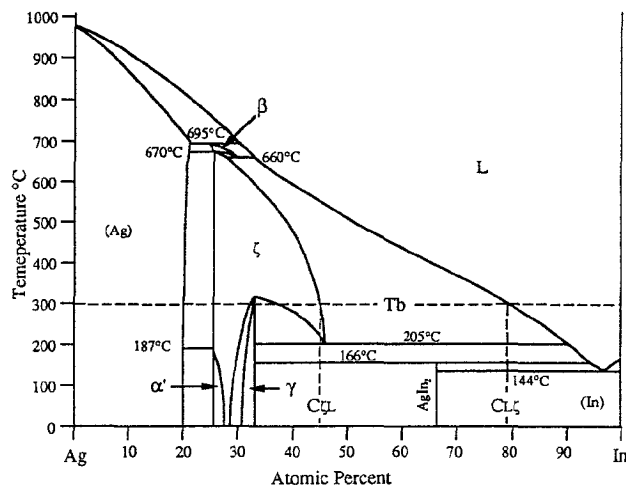

Figure 8: Silver-Indium phase diagram. Bonding at 300°C will result in the formation of ζ and some γ. These intermetallic phases will grow as more copper is dissolved by the indium. Eventually the intermetallic phase will bridge the joint resulting in a semi-solid bond at temperature.

of three. The titanium foils were also eutectic bonded using a copper interlayer. However, an interaction zone of about 1μm (40μin) developed at the interface. This process was designed to fabricate structural components for the space shuttle.

The LID bonding process described by Norris [21] uses a copper-nickel foil to bond engine ducts made of thin gauge titanium. Static tensile tests on thick sections indicated that parent metal strengths are achieved provided the faying surface roughness is "fine". Bonding pressure was provided by delta-alpha tooling which relies on the difference in thermal expansion between the parent material and components of a strongback bonding tool. This process is unique in that the interlayer liquifies on its own at the bonding temperature hence it is a type II process, but the parent metal is not a component of the interlayer.

The NOR-TI-BOND process [13,14] required electroplated copper to TLP bond titanium structural sections. Local resistance heating provided by rollers liquified the joint which then solidified on cooling. A subsequent heat treatment lowered the copper content in the joint from 70% to below 7%. Residual copper in the joint affected the ß-α transformation kinetics and the joint microstructure with a corresponding effect on the mechanical properties. While parent metal shear strengths were obtained over a range of temperatures, bend deflection and fracture toughness were susceptible to structure and residual copper content. The maximum copper content had to be kept below 7% to meet the fracture toughness requirement.

Summary

The TLP process has been applied to many systems. For fast processing times, an interstitial diffuser in a thin foil near the liquidus composition should be selected. Selection criteria for the applicability of TLP bonding to a particular system include stability at bonding temperature coupled with a solubility for

the MPD. Intermetallic formation can be tolerated in binary systems, but may require extended homogenization. In ternary and higher order systems, intermetallics should be avoided. Diffusivities on the order of $10^{-8} cm^2/sec$ or higher are needed to achieve reasonable bonding times. Superalloys are amenable to this process because of their intrinsic stability at high temperatures, and their ability to diffuse interstitials without detriment to mechanical properties. Parent metal mechanical properties are achieved but there always remains some susceptibility of the joint to reduced toughness.

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